

Dry and thin film typed delivery system for whitening teeth

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Abstract: KR 2003059552 - A NOVELTY - A system for delivering a *tooth* *whitening* substance to a plurality of adjacent *teeth* is provided, wherein the *tooth* *whitening* substance is dispersed within a polymer. Therefore, the system is not sticky when applied, does not need to be removed after use thereof and is conveniently used. DETAILED DESCRIPTION - A thin film typed delivery system contains 5-80% by weight of a polymer, 0.1-50% by weight of a *tooth* *whitening* substance and additionally a protective film layer (2) with a thickness of 5-150 micro-m. The polymer has an adhesion property to the teeth when hydrated by saliva, but no adhesion property when stored. The polymer is hydroxypropylmethyl cellulose, hydroxyethyl cellulose, polyvinyl alcohol, polyvinylpyrrolidone, hydropropyl cellulose, carbomer, a copolymer of methyl vinyl ether and maleic anhydride and/or polyethylene oxide. The *tooth* *whitening* substance is hydrogen peroxide and/or carbamide peroxide.

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(54) Dry thin film-type tooth whitening material delivery system.

Summary

This invention applies to a dry thin film-type tooth whitening material delivery system. In detail, this tooth whitening material delivery system employs a dry thin film of macromolecules in which the active whitening material is dispersed. These macromolecules do not have adhesion power when stored. They obtain adhesion power when they are hydrated by saliva from teeth upon application. While gel-type tooth whitening material is sticky and slippery when it is applied on teeth, the aforementioned thin film-type tooth whitening material has the advantage of being free from these problems. The thin film-type tooth whitening material obtains adhesive power only when it is exposed to moisture. Additionally, the protective film layer that can be applied on the dry thin film protects the active whitening material to last long enough for whitening action by preventing the saliva from attacking the film in the mouth. It is not necessary to remove

the film after use since the saliva dissolves it after a certain time. This is a very convenient tooth whitening material delivery system.

Representative figure

Figure 1

Key words

Tooth whitening, thin film-type, polyethylene oxide, protective film layer

Details

Simple explanation of figures

Fig.1 shows one example of a tooth whitening material delivery system that consists of tooth whitening material film and protective film layers according to this invention.

Fig. 2 shows one example of a tooth whitening material delivery system that consists of a whitening material, a protective film and adhesion help layers according to this invention.

Fig. 3 shows a graph of tensile strength versus time from Example 5 according to this invention.

<Explanation of the critical components in figures>

1: Protective film layer 2: Whitening material layer

3: Adhesion help layer

Detailed explanation of invention

Purpose

Technology related to invention and conventional technology

This invention applies to a dry thin film-type tooth whitening material delivery system. In detail, this tooth whitening material delivery system employs a dry thin film of macromolecules in which the active whitening material is dispersed. These macromolecules do not have adhesion power when stored. They obtain adhesion power when hydrated by saliva from teeth upon application. While gel-type tooth whitening material is sticky and slippery when it is applied on teeth, the aforementioned thin film-type tooth whitening material has the advantage of being free from these problems. The thin film-type tooth whitening material obtains adhesive power only when it is exposed to moisture. Additionally, the protective film layer that can be applied on the dry thin film protects the active whitening material to last long enough for whitening action by preventing the saliva from attacking the film in the mouth. It is not necessary to remove the film after use since the saliva dissolves it after a certain time. This is a very convenient tooth whitening material delivery system.

A tooth consists of an inner layer of dentin and outer layer of protective enamel. Generally, the tooth enamel is opaque white or slightly yellow. In its natural state, the enamel has space or holes. When coloring or discoloring materials enter the space or holes, the teeth change color. Many materials that individuals

encounter daily may cause coloring or reduce the whiteness of the teeth. In particular, when food and tobacco accumulates on the enamel, it forms a thin film. These coloring and discoloring materials can pass through the enamel layer. If this has continued for many years, then the enamel may have become discolored. Certain diseases or environmental factors may cause discoloring of the teeth.

Even though discoloring of the teeth does not indicate ill health or causes any health problems, bright and white teeth have been desired by individuals from many reasons.

Fluoride tooth paste is one of tooth whitening material. By chemical reaction, it removes food pieces, and stains on the teeth surface from nicotine, coffee and tea, and blotches caused by coloring of the tartar and soft precipitate on the teeth surface. In general, it is used for refreshing and reducing odor of the mouth. However, it has been difficult to accomplish whitening of the teeth only using tooth paste and a tooth brush.

Consequently, dentists have offered two professional teeth whitening programs: in-clinic and outpatient. The in-clinic program begins generally by preparation of a rubber dam to protect the soft tissue within the mouth from the hydrogen peroxide that is the whitening material. The patients who are in the outpatient program administer the weak whitening material themselves on the teeth for a long period of time, generally several hours each day for several weeks. It is necessary for the patients who are in the in-clinic program that a special device has been prepared for each patient that fits his or her teeth. This device delivers whitening gel to the teeth. However, these methods are expensive and inconvenient for daily life.

US Patent 5,310,563 suggests that use of a pressurized correcting putty on the teeth to solve the aforementioned problems, but with gel type of whitening material, some is left on the hand and is very sticky when it is applied on teeth. A special mouth piece is required for applying gel-type whitening material on the teeth. Wearing mouth piece causes inconvenience for daily life.

US Patent 5,425,953 suggests a liquid whitening polymer composite. This patent takes advantage of the property of the polymer. This liquid polymer composite is liquid before being applied on teeth, but it turns into a solid film upon being applied on teeth. This is the way to correct the problem of gel-type whitening material. But it takes time for the polymer to solidify. It is sticky until the polymer solidifies. Furthermore, it has the disadvantage of being hazardous to health since it employs organic solvent.

US Patent 5,891,453 employs a system that delivers whitening material to the teeth via a strip. This strip has low [illegible]. This delivery system is convenient for daily life, but it is inconvenient since the waterproof strip has to be removed after a certain time. By conditioning the film at the time of application, the film can be applied easily. However, it contains foreign substance as the waterproof material and is sticky and leaves residues on the hand just like gel-type whitening material.

Technological Purpose of Invention

The makers of this invention solved the aforementioned problems and have achieved a dry thin-film type tooth whitening material delivery system. In this system, the whitening material is dispersed in a macromolecule compound that does not have adhesion power during storage, but gains adhesion power upon hydration by saliva on the teeth.

Thus, this invention aims to remove stickiness from the hand and slipperiness on teeth when the whitening material is applied.

Framework and Reaction of Invention

This invention is characterized by a tooth whitening material delivery system. This system employs a dry thin film that is made of a macromolecular compound in which the tooth whitening material is dispersed. This macromolecular compound gains adhesion power on teeth only when it is hydrated.

The detailed explanation of the invention is as follows:

This invention applies to a tooth whitening material delivery system. This system employs a dry thin film that is made of a macromolecular compound in which the tooth whitening material is dispersed. This macromolecular compound gains adhesion power on teeth only when it is hydrated.

The components in the tooth whitening material delivery system are explained in detail in the following passages.

As for the adhesive macromolecule compound that gains adhesion power upon hydration in the tooth whitening material delivery system, hydroxypropylmethylcellulose, hydroxyethylcellulose, polyvinyl alcohol, polyvinylpyrrolidone, hydroxypropylcellulose, carbomer copolymer from methylvinylether and maleic anhydride and polyethylene oxide may be used. The concentration of the adhesive macromolecular compound is desired to be 5~80% by weight in the tooth whitening material delivery system. 5~60% by weight is better. If the concentration is below 5% by weight, the whitening material layer has very little adhesion and it may run off and will not stick to the teeth. If the concentration is above 80% by weight, the degree of adhesion is too high for it to work. Polyethylene oxide of average molecular weight of 100,000~4000,000 is desired. A molecular weight of 100,000~2000,000 is better. If the average molecular weight is below 100,000, it is difficult to attain the proper degree of adhesion. If the average molecular weight is above 400,000, the adhesion degree is very high even at the low concentration.

As for the tooth whitening active material, peroxide, chlorous acid metal, perborate salt, percarbonate salt, peroxide acid, or mixture of these may be used. The concentration of the tooth whitening material in the delivery system is desired to be 0.1~50 % by weight. 0.5~30 % is better. If the concentration of the tooth whitening material is below 0.1%, the desired whitening cannot be obtained. If the concentration of the tooth whitening material is above 50%, it may cause corrosion of the teeth surface and damage of the mouth's mucous membrane, which will create a safety problem. As the above mentioned peroxide, hydrogen peroxide, calcium peroxide, carbamide peroxide, or a mixture of these peroxides may be used. Hydrogen peroxide and carbamide peroxide are more desirable. The metal chlorous acid include calcium, barium, magnesium, lithium, sodium and potassium salt.

The tooth whitening material delivery system in this invention contains plasticizer, stabilizer and pyrophosphate.

The aforementioned plasticizer is used for the formation of the dry thin film of the tooth whitening material delivery system. The concentration of plasticizer to adhesive polymer is desired to be 5.0~50 % by weight. If the concentration of the plasticizer is below 5.0%, pliability decreases. If the concentration of the plasticizer is above 50%, the stability of the manufactured film decreases. The general film plasticizer: polyethylene glycol, propylene glycol, glycerine and triethylcitrate may be used for the plasticizer in this system.

The aforementioned stabilizer is used for the improvement of the tooth whitening active material stability. The concentration of the stabilizer is desired to be 0.01~5.0 % by weight in the tooth whitening material delivery system. The chelating agent and antioxidation agent may be used for the stabilizer.

Ethylenediaminetetraacetic acid and its salt, sodium stannate and potassium stannate may be used for the chelating agent. Butylated hydroxytoluene, nordihydroguaiaretic acid, propyl gallate, trihydroxybutyrophenone and butylated hydroxyanisole may be used for the antioxidation agent.

The aforementioned pyrophosphate is effective in removing tar and chelating metal compounds. It is used for preventing tartar in this invention of the tooth whitening material delivery system. The concentration of the pyrophosphate is desired to be 1.0~10.0 % by weight in the tooth whitening material delivery system. Dialkali metal pyrophosphate and tetrametal pyrophosphate may be used. For example: $\text{Na}_4\text{P}_2\text{O}_7$, $\text{K}_4\text{P}_2\text{O}_7$, $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$, and $\text{K}_2\text{H}_2\text{P}_2\text{O}_7$.

The pH adjuster, sweetener and flavorings may be added as general additives to the tooth whitening material delivery system in this invention besides the aforementioned components.

The pH adjuster adjusts the pH of the tooth whitening material delivery system at pH 4.0~11.0 for the stability during long storage. NaH_2PO_4 , Na_2HPO_4 , sodium pyrophosphate, potassium pyrophosphate, sodium carbonate, potassium hydroxide, ammonium hydroxide, triethanol amine, citric acid and sodium citrate may be used as the pH adjuster.

Sweetener and flavorings may be added to the tooth whitening material delivery system for its convenient use.

Sorbitol, mannitol, xylitol, steviol glycoside(?) and saccharine may be used for the sweetener. General food flavorings may be used in this invention.

Ethanol, water, or mixture of ethanol and water (9:1~6:4 v/v %) may be used as solvent for the aforementioned additives.

This invention includes an additional layer of protective layer to prevent the dry thin film tooth whitening active material from adhering to the mouth's mucous membrane. Fig. 1 shows this. It can be explained in detail with Fig. 1 as follows. The aforementioned protective layer (1) acts as a protective wall so that the saliva will not make contact with the active tooth whitening material and dissolve the film. It prevents the active whitening material from being activated by the lip, tongue and other soft tissue on the surface of the teeth. Also it prevents the tooth whitening active material layer (2) from adhering on the other side of the teeth. It is desired for the tooth whitening material active layer to dissolve and be activated slowly so that the activity on the teeth can be lengthened and maximized.

The aforementioned protective layer uses a water soluble macromolecule compound that dissolves in water at pH 5.0 – 8.0 at 37° C. The aforementioned water soluble macromolecule compound is dissolved by saliva after it is activated on the teeth and whitens the teeth. Therefore, it is not necessary to remove the active film layer. The soft film layer gives a nice feeling. One or two compounds among hydroxypropylpropylmethylcellulose phthalate, eudragit(?) and cellulose acetate phthalate may be used. Hydroxypropylmethylcellulose phthalate is good.

The aforementioned protective film layer does not adhere to the mouth's mucous membrane. Therefore, it can make contact with nearby mouth tissue without any irritation. Since this protective film layer disappears by dissolving in saliva after certain time from use, it is convenient not to worry about removal of it.

Also, the aforementioned protective film layer should have a thickness of 5~150 μm , which causes less skin irritation. If the thickness is less than 5 μm , the tooth whitening active material may cause skin irritation. If the thickness is more than 150 μm , it gives an uncomfortable feeling of having a foreign substance in the mouth. The thickness of the tooth whitening active material (2) with the protective film layer is desired to be 50~500 μm . If the thickness is less than 50 μm , it is too soft for handling. If the thickness is more than

500 μm , it is hard to make due to problems in drying, and has the disadvantage of having the feeling of having a foreign substance in the mouth.

This invention also applies to a tooth whitening material delivery system that has an adhesive help layer (3) in addition to the tooth whitening active material layer and the protective film layer as Fig. 2 shows. The aforementioned adhesive help layer (3) helps the tooth whitening active material layer adhere on the teeth surface. The previously mentioned macromolecule compound that gains adhesive power upon hydration may be used for this layer.

As described above, the tooth whitening material delivery system of this invention uses a compound that gains adhesion power only when it is hydrated. This is different from the gel-type tooth whitening material that is sticky and slippery when it is applied. The protective film that can be laminated on the dry thin film of tooth whitening active layer makes it possible for the dry thin film to be attacked by saliva slowly, which gives enough time for the active tooth whitening material to work. Since this system is eventually dissolved by saliva, it has the benefit of not having to be removed after its use.

Below, this invention is explained through practical examples, though the invention is not limited to only these.

Experiment 1

<Production of the protective film layer>

Hydroxypropylmethylcellulose phthalate (HPMCP), 20% by weight is dissolved in acetone, 73% by weight. Triacetin 7% by weight is mixed in the above solution. After air bubbles are removed, the mixture is spread with thickness of 150 μm . It is dried at 50°C with hot air to make the protective film layer.

<Production of the whitening material layer>

The composition of the components is listed in Table 1. Polyethyleneoxide, polyethylene glycol disodium pyrophosphate (DSPP) and sodium stannate is mixed. The mixture is dispersed in ethanol. Liquid sorbitol and hydrogen peroxide are added to the dispersion. The pH was adjusted to 4.5 ± 0.5 . The air bubble is removed.

The mixture is spread with thickness of 300 μm . It is dried at 50°C with hot air to make the whitening material layer.

<Production of the tooth whitening material delivery system>

The above protection film layer and the whitening material layer were laminated and then cut to 1.5 X 6.5 cm rectangles to make the tooth whitening material delivery system.

Experiment 2 – 5

The protective film layer was produced in the same way as described in Experiment 1, and then the protective film layer was prepared with thickness according to Table 1. The whitening material layer was produced in the same way as described in Experiment 1, and the whitening material layer was prepared with the thickness according to Table 1.

The above protection film layer and the whitening material layer were laminated to make the tooth whitening material delivery system.

			Experiment (weight %)				
			1	2	3	4	5
Whitening material layer (weight %)	Tooth whitenin g active material	hydrogen peroxide	15	15	15	-	-
		carbamide peroxide	-	-	-	30	30
	Macro- molecule	PEO (Mw 200,000)	-	20	-	-	-
		PEO (Mw 300,000)	-	-	-	10	10
		PEO (Mw 900,000)	7.5	-	7.5	-	-
	Plasticize r	PEG 1,000	1.5	4	1.5	3	5
	Stabilizer	Sodium stannate	0.1	0.1	0.1	0.1	0.1
	Pyrophosphate		1.5	1.5	2.0	2.0	2.0
	Sweetene r	Sorbitol	10.5	10.5	10.5	10.5	10.5
	pH adjuster	Phosphoric acid	Correct amount	Correct amount	Correct amount	Correct amount	Correct amount
	Solvent	Ethanol	63.9	48.9	63.4	44.4	42.4
	Whitening material layer (μm)			300	500	300	300
Protective film layer (μm)			150	100	50	50	50

Comparison experiment

This invention has been compared to White Strips (Manufacturer: Pamp; G Corp., USA) tooth whitening material delivery system that is available.

Test Experiment 1

The effect of the whitening action has been compared with the dry thin film tooth whitening material delivery system from Experiment 5 by comparison experiment through the following method:

40 healthy adult volunteers who are fit for four weeks of clinical test have been selected. This group is divided into two groups: A and B, 20 persons each. The White Strips comparison study system was applied to A Group. The whitening material delivery system from Experiment 5 was applied to B Group. The application area of the testing system was the upper teeth. The color baseline of the teeth was measured and set by VITA LUMIN Vacuum Fabraskala Shade Guide for each volunteer. The application method of the White Strips was taken for the test. The whitening material delivery system was applied to the teeth for 30 minutes once a day. Using other tooth whitening material was avoided. After four weeks, the teeth color was measured by the VITA LUMIN Vacuum Fabraskala Shade Guide for each volunteer and changes from the base line were recorded. The effect of the tooth whitening material has been evaluated by the change of the teeth color. The results are listed in Table 2.

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[Table 2]

Distribution of shade-change for each group

Change in color value	Comparison experiment (Group A)	Experiment 5 (Group B)
No change	0	0
1~3	3	4
4~6	5	6
7~9	8	6
10~12	4	4
13~15	0	0

To examine if there is an improvement for each tooth whitening system, the statistical method, t-test was used with the data in the above table. The results are summarized in Table 3.

[Table 3]

Comparison of degree of improvement for each group (t-test)

Comparison Experiment (Group A) (Average : standard deviation)	Experiment 5 (Group B) (Average : standard deviation)	P-Value	Significance level
6.85 : 3.08	6.55 : 3.07	0.16	0.05

As Table 3 shows P-Value, 0.16 is greater than significance level, 0.05. This proves that the effect of the whitening is same for both groups.

Test Experiment 2: The rate of dissolution of the film in the mouth.

The rate of dissolution of the two films from the comparison experiment and Experiment 5 in the artificial saliva (pH 7.0, 37°C) has been measured. The results are listed in Table 4.

[Table 4]

Rate of dissolution of the film (unit: min.)	
Comparison experiment	Does not soluble in the mouth

Experiment 5

Within 40 min.

As the above Table 4 shows, the tooth whitening active material from this invention does not dissolve while the whitening material is active (30 minutes) but dissolve after the action is completed. It proves that this invention has advantages. It is not necessary to remove the film.

Test Experiment 3. Convenient Factors of the Product

To measure the feeling of foreign substances in the mouth when the whitening film was applied, films from the comparison experiment and Experiment 5 were applied on the upper teeth of the test volunteers. The results are listed in Table 5.

[Table 5]

	Feeling of presence of foreign substances at application	Stickiness on the hand before application
Comparison experiment	Severe	Very sticky
Experiment 5	None	None

This Table indicates that the film from this invention does not leave adhesive material on the hand and does not cause stickiness and does not give an uncomfortable feeling foreign substances being present. Therefore, it is convenient to use this invention system.

Test Experiment 4: The stability of the product 1.(Major component)

The films from the comparison experiment and Experiment 5 were stored at 4°C, 25°C, room temperature and 40°C.

The level of hydrogen peroxide and carbamide peroxide was measured at the beginning of the storage and after six weeks. Hydrogen peroxide was measured by KMnO₄ method and carbamide peroxide was measured by iodine method. The results are listed in Table 6.

[Table 6]

	Comparison experiment (%)		Experiment 5 (%)	
	Beginning	6 weeks	Beginning	6 weeks
Cooling (4°C)	101.1	99.8	100.8	100.3
Room Temp (25°C)	101.1	99.9	100.8	100.5
Heat (40°C)	101.1	87.4	100.8	90.4

As the above table indicates, the film from this invention has superior temperature stability compared to the gel-type since it is a dry film type.

Test Experiment 5: The stability of the product 2 (Tensile strength test)

The film of this invention is dissolved in saliva and disappears. Due to this property the tensile strength of the product may deteriorate by minor changes unlike the waterproof strip. The decrease of the tensile strength may decrease the quality of the product. The tensile strength of the film from Experiment 5 was measured at time intervals. The results are listed in Fig. 3.

The tensile strength was measured for the samples that were stored at 4°C, 25° C, room temperature and 40°C before storage and after six weeks by QTS Texture Analyzer.

As Fig. 3 shows, the tensile strength of the film from Experiment 5 gave a constant value after the maturing period. This indicates that the product keeps its form.

Result of Invention

The tooth whitening material delivery system in this invention is free from stickiness and leaves no residual material on the hand when it is applied. When the protective film is laminated on the tooth whitening layer, the active material is activated slowly and lasts long enough for whitening. Since it dissolves in the mouth, removal of it is not necessary.

It is convenient to use and does not give any uncomfortable feeling of the presence of foreign substances. It does not interfere with speech and appearance. It is a useful system for tooth whitening.

(57) Scope of application

Claim 1

A tooth whitening material delivery system is characterized by a tooth whitening material delivery system that employs a dry thin film of macromolecule compounds that gains adhesion power upon hydration. The tooth whitening material is dispersed in this macromolecule compound.

Claim 2

A tooth whitening material delivery system characterized by a concentration of the macromolecule compounds in the thin film mentioned in Claim 1 that is 5 ~80 % by weight, and a concentration of the tooth whitening active material that is 0.1~50% by weight.

Claim 3

A tooth whitening material delivery system characterized by use of hydroxypropylmethylcellulose, hydroxyethylcellulose, polyvinylalcohol, polyvinylpyrrolidone, hydroxypropylcellulose, carbomer, copolymer of methylvinylether and maleic anhydride and polyethylene oxide, or a mixture of two selections from this list, for the macromolecule compounds in Claim 1 and Claim 2.

Claim 4

A tooth whitening material delivery system characterized by an average molecular weight of the aforementioned polyethyleneoxide in Claim 3 at 100,000~4,000,000.

Claim 5

A tooth whitening material delivery system characterized by having the tooth whitening active material in Claim 1 and Claim 2 being hydrogen peroxide, carbamide peroxide, or a mixture of these.

Claim 6

A tooth whitening material delivery system characterized by having the protective film layer in Claim 1 laminated on the dry thin film so the tooth whitening active material will not make contact mouth's mucous membrane.

Claim 7

A tooth whitening material delivery system characterized by having hydroxypropylmethylcellulose phthalate, hydroxypropylmethylcellulose acetate succinate, eudragit(?), cellulose acetate phthalate, or a combination of two used for the protective film layer in Claim 6.

Claim 8

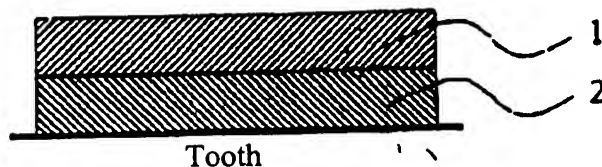
A tooth whitening material delivery system characterized by having hydroxypropylmethylcellulose phthalate as the protective film layer in Claim 7.

Claim 9

A tooth whitening material delivery system characterized by having the thickness of the protective film layer in Claim 6 be 5 ~150 μm .

Figure

Fig. 1



FigF. 2

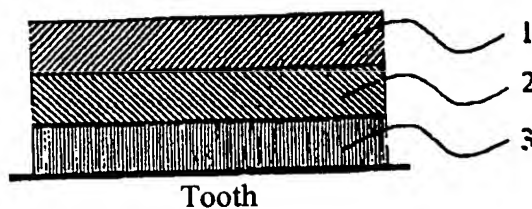
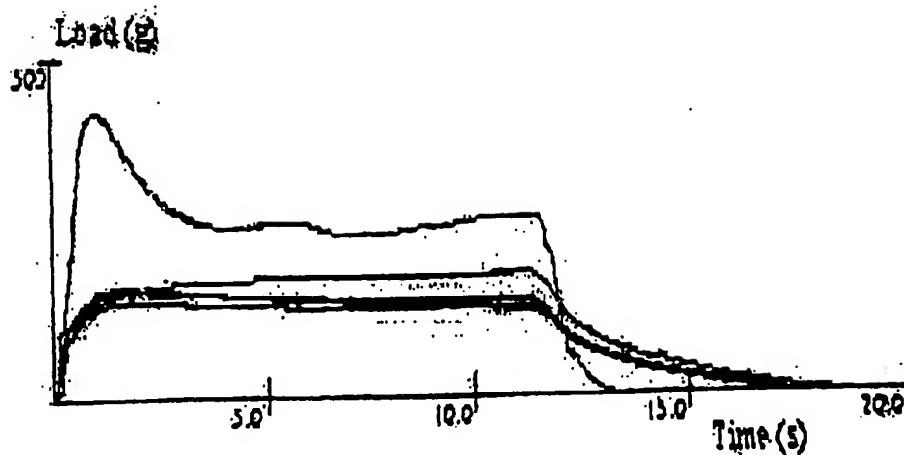


Fig. 3



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